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A claim to priority pursuant to 35 U.S.C. § 119 of European Patent Application No. 02080128.8 filed December 6, 2002 was previously made. To complete the claim a certified copy of the European Patent Application is enclosed.

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The attached documents are exact copies of the European patent application described on the following page, as originally filed.

Les documents fixés à cette attestation sont conformes à la version initialement déposée de la demande de brevet européen spécifiée à la page suivante.

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Der Präsident des Europäischen Patentamts;  
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets  
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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:  
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.  
If no title is shown please refer to the description.  
Si aucun titre n'est indiqué se referer à la description.)

Improved cyclic ketone peroxides compositions

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## IMPROVED CYCLIC KETONE PEROXIDES COMPOSITIONS

The present invention relates to peroxide compositions comprising cyclic ketone peroxide and phlegmatizer. It also pertains to the use of such 5 compositions in (co)polymerization and polymer modification processes.

Such peroxide compositions are disclosed in WO 98/33770, wherein formulations of cyclic methyl ethyl ketone peroxide in mixtures of Primol® 352 and isododecane are described. Although these formulations have acceptable 10 safety properties, the active oxygen content is still relatively low. It is furthermore shown in Comparative Examples A and B that the single phlegmatizer cyclic ketone peroxide formulations in either Primol® 352 or isododecane are not safe.

15 In US 6,358,435 peroxide compositions comprising cyclic ketone peroxide and a phlegmatizer are also disclosed. The phlegmatizer is characterized by a 95% boil-off point falling in the range of 220-265°C. These peroxide compositions may have a relatively high active oxygen content which are generally safe and storage stable at 20°C.

20 However, the inventors have surprisingly found that compositions of the cyclic ketone peroxides having such a high active oxygen content, impair a safety hazard when stored at -10°C or lower due to the formation of explosive crystals. More specifically, a concentrated composition comprising a cyclic ketone 25 peroxide e.g. cyclic methyl ethyl ketone peroxide, which is the peroxide of the Examples disclosed in US 6,358,435, forms crystals upon storage at -10°C, which results in an explosion when heated and thus impairs an enormous safety hazard.

30 It is an object of the present invention to alleviate the above-described problems and improve the safety and storage stability of the conventional

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compositions comprising cyclic ketone peroxides which form crystals when stored at -10°C.

The present invention provides a cyclic ketone peroxide composition 5 comprising one or more crystallizing peroxides, optionally one or more conventional phlegmatizers (diluents), and a co-crystallizing compound which solidifies in the final formulation at a solidification temperature above the crystallization temperature of the crystallizing peroxide.

Preferably, the solidification of the co-crystallizing compound is below room 10 temperature (i.e. 20°C) so that the formulation is a liquid at conventional handling temperatures. However, if it is desired to work with a waxy or solid product, a co-crystallizing compound with a higher solidification temperature can be used.

The composition of the invention will not explode when heated after storage at 15 temperatures of -10°C or even lower. With the use of the co-crystallizing compound in the composition, it is ensured that the solid co-crystallizing compound is already present at a temperature which is higher than the temperature at which the peroxide crystallizes in conventional formulations, thus preventing the formation of peroxide crystals as formed in conventional 20 formulations.

Typically, when the composition is cooled to a temperature below e.g. 20°C, the viscosity of the co-crystallizing compound (e.g. a paraffin having a melting point exceeding -5°C) first increases, whereby the composition becomes more 25 viscous, preferably thixotropic. When the peroxide composition is further cooled to a storage temperature of -10°C or even lower, the composition preferably becomes a solid or a highly viscous material such as a gel.

According to a non-binding theory, the formation of peroxide crystals is 30 prevented by spreading of the cyclic ketone peroxide throughout the dispersed

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co-crystallizing compound of the invention. This spreading can be achieved by either adsorption of the peroxide into the co-crystallizing compound, or by isolating small quantities of the peroxide into the compound. With adsorption is meant the uptake of molecules of the cyclic ketone peroxide. When larger clusters or even small volumes are located inside cavities of similar sizes, but which are smaller than the peroxide crystals formed in conventional formulations, we speak of isolation. In the case of isolation, it is envisaged that in the cavities of the solidified co-crystallizing compound small solids of the peroxide are present, which may either comprise the peroxide or be a mixture of the phlegmatizer and the peroxide.

The spreading of the cyclic ketone peroxide throughout the solidified co-crystallizing compound is expected to bring about the presence of only volumes of peroxide of which only a small amount (or none at all) are in contact with or close to one another. The size of these volumes is so small that the explosion risk is reduced to a desired level so as to ensure the appropriate safety and storage stability of the composition.

The invention is particularly suitable for compositions having a high concentration of the cyclic ketone peroxide, e.g. compositions that are saturated or supersaturated at -10°C. Consequently, according to the invention new compositions with a high active oxygen content can be prepared, which are safe and storage stable, even at a very low temperature.

The terms "storage stability" or "storage stable" used in this specification mean that the composition is not liable to explode when stored or heated after storage at a storage temperature of -10°C or lower.

The term "crystallizing peroxide" is used to denote a cyclic ketone peroxide which, when blended with the conventional phlegmatizer (diluent) shows formation of crystals at a temperature, hereinafter called the crystallization temperature, above a temperature of -20°C.

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The term "solidification" of the co-crystallizing compound refers to the process of decreasing the temperature of the peroxide composition to the solidification temperature at which the viscosity of the co-crystallizing compound is increased in the composition to such an extent that the co-crystallizing compound 5 separates from the peroxide composition and becomes highly viscous or solid.

The co-crystallizing compound can be any suitable compound which separates from the final peroxide composition, preferably by the formation of solid particles at a temperature above the crystallization temperature. Preferably, 10 these compounds separate or form solid particles at a temperature which is at least 5°C above the crystallization temperature, more preferably at least 10°C above the crystallization temperature, and most preferably at least 20°C above the crystallization temperature. The compound is preferably chosen such that the peroxide composition is liquid at either the recommended storage 15 temperature of the composition or the handling temperature when the composition is used in e.g. polymerization processes, whichever temperature is lowest.

The co-crystallizing compound is preferably a hydrocarbon with or without 20 hetero atoms like oxygen, nitrogen or sulphur, for example. Most preferred are hydrocarbons without hetero atoms. The co-crystallizing compound can be selected from cyclic and non-cyclic hydrocarbons, esters, ester phosphate, cellulose esters, hydrogenated castor oil and fumed silica. Examples of cyclic and non-cyclic hydrocarbons are Paraffin (ex Mallinckrodt Baker B.V.), TerHell 25 5205 (ex Schümann-Sasol), Norpar 15 (ex Exxon), n-hexadecane, n-eicosane, n-eneicosane, octadecane, tricyclohexylmethane, or aromatic hydrocarbons such as naphtalene, 1,2,4,5-tetramethylbenzene, 1,4-dihydronaphtalene, 3-methylnaphtalene, hexamethylbenzene, biphenyl, diphenylmethane, 1,2-diphenylmethane, 9-methylfluorene, phenatrene, 9,10-dihydrophenatrene, 30 1,2,3,4-tetrahydrophenatrene, and octahydroanthracene. Examples of esters

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are dicyclohexylphthalate, methylpalmitate,  $\alpha$ -naphthylacetate,  $\beta$ -naphthylacetate, phenylbenzoate, ethyl diphenylacetate, dimethyloxalate, trimethylene carbonate, pentamethylene carbonate, hexamethylene carbonate, methylacetyl salicilate, dimethyl phenylmalonate, methyl p-vinylbenzoate and methylhydrogen succinate. Examples of ester phosphates are triethyl phosphate, tricresyl phosphate, trixylyl phosphate, cresyl diphenyl phosphate, 2-ethylhexyl-diphenyl phosphate, isodecyl-diphenyl phosphate, tri(2-ethylhexyl) phosphate, dimethyl methyl phosphonate, chlorinated phosphate esters, tributyl phosphate, and tributoxyethyl phosphate. Examples of cellulose ester are the reaction products of cellulose and acid compounds selected from, for example, acetic acid, propionic acid, butyric acid, phthalic acid, trimellitic acid and mixtures thereof. Examples of commercially available hydrogenated castor oils are Rheocin® (ex. Sud-Chemie), Thixcin® (ex. Rheox Inc.), and Luvotix® (ex Lehmann & Voss). Examples of commercially available fumed silica include Aerosil® (ex. Degussa), Cab-o-Sil® (ex. Cabot) and HDK® (ex. Wacker Chemie).

Preferably, the co-crystallizing compound is a non-cyclic hydrocarbon, more preferably a straight chain hydrocarbon such as Paraffin (ex Mallinckrodt Baker B.V.), TerHell 5205 (ex Schümann-Sasol), Norpar 15 (ex Exxon), n-hexadecane, n-eicosane, n-eneicosane, and octadecane.

Preferably, the peroxide composition according to the invention comprises at least 0.5 wt%, more preferably at least 1 wt% and most preferably at least 2 wt%, and preferably at most 99 wt%, more preferably at most 80 wt%, and most preferably at most 70 wt% of the co-crystallizing compound, based on the total weight of the composition.

Typically, the co-crystallizing compound is chosen so that it does not adversely affect the polymerization process in which the cyclic ketone peroxide composition is used. The co-crystallizing compound may take part in the

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process wherein the final composition is used, whereby it is preferably incorporated in the polymer. Preferably, the peroxide compositions of the invention are suitable for use in polymer-based products which are food approved.

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For determining whether the cyclic ketone peroxide composition comprises a crystallizing peroxide which can be formulated according to the present invention (i.e. which is liable to explode when heated after storage at temperatures as low as -20°C or below), a test was developed with which it is possible to establish whether crystals are formed at a temperature above -20°C. To a composition of just a cyclic ketone peroxide and isododecane (as a conventional phlegmatizer) in the same weight ratio as used in the composition under investigation, seeds of the pure cyclic ketone peroxide were added, after which the composition was stirred for 24 hours at -20°C. If crystals of the cyclic ketone peroxide are formed at this temperature, the peroxide was considered to be a crystallizing peroxide which can be formulated according to the invention.

Examples of cyclic ketone peroxides that can crystallize in conventional formulations are the peroxides derived from acetone, acetyl acetone, methyl ethyl ketone, methyl propyl ketone, methyl isopropyl ketone, methyl butyl ketone, methyl isobutyl ketone, methyl amyl ketone, methyl isoamyl ketone, methyl hexyl ketone, methyl heptyl ketone, diethyl ketone, ethyl propyl ketone, ethyl amyl ketone, methyl octyl ketone, methyl nonyl ketone, cyclopentanone, cyclohexanone, 2-methylcyclohexanone, 3,3,5-trimethyl cyclohexanone, and mixtures thereof.

The composition may also include mixtures of the cyclic ketone peroxide with a (cyclic) ketone peroxide or any other peroxide which is not in accordance with the present invention. Decisive for the final composition is whether any crystal growth is observed in the test as described above.

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Preferably, the composition of the invention comprises at least 1 wt%, more preferably at least 5 wt%, and most preferably at least 10 wt% of the cyclic ketone peroxide, and preferable at most 99 wt%, more preferably 90 wt%, and most preferably 80 wt% of the cyclic ketone peroxide, based on the total weight  
5 of the composition.

Preferably, the invention composition has an active oxygen content of more than 3 percent by weight of oxygen (wt%), based on the total weight of the peroxide composition. Preferably, the composition comprises at most 12 wt%,  
10 more preferably at most 10 wt%, and most preferably at most 8 wt% of oxygen, based on the total weight of the peroxide composition. For storing and transporting bulk quantities of the invention composition it may be necessary to dilute the composition with the phlegmatizer of choice thereby reducing the active oxygen content of the cyclic ketone peroxide composition.  
15

The phlegmatizer in the composition of the present invention can be any suitable phlegmatizer which is not identical to the co-crystallizing compound, or a mixture of one or more of such phlegmatizers.

20 Examples of the phlegmatizer include linear and branched hydrocarbon solvents such as tetradecane, tridecane, Isopar® M, Exxsol® D80, Exxsol® D100, Exxsol® D100S, Soltrol® 145, Soltrol® 170, Varsol® 80, Varsol® 110, Shellsol® D100, Shellsol® D70, Halpasol® i 235/265, and mixtures thereof. Particularly preferred phlegmatizers are Isopar® M and Soltrol® 170. Examples  
25 of other suitable phlegmatizers can be found in US 5,808,110. Although less preferred, it is also possible to use a specific fraction of the styrene oligomers of WO 93/25615.

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Preferably, the peroxide composition according to the invention comprises at least 1 wt%, more preferably at least 5 wt%, and most preferably at least 10 wt% of the phlegmatizer, and preferable at most 99 wt%, more preferably 90 wt%, and most preferably 80 wt% of the phlegmatizer, based on the total weight 5 of the composition. It is noted that the phlegmatizer and co-crystallizing compound can be combined before use. Hence, it is feasible to purchase and use specific phlegmatizers that contain co-crystallizing species. Decisive for whether or not a co-crystallizing compound according to the present invention is present in the formulation is the separation of one or more species at a 10 temperature above the crystallization temperature of the peroxide.

The safety of the compositions of the present invention were determined with a specifically developed test with which the so-called crystallization point of the peroxide is determined. By determining this crystallization point, it can be 15 established whether crystals are formed comparable to the crystals formed in conventional formulations (without co-crystallizing compounds) of this peroxide. The crystallization point is defined as the temperature at which the crystals – which are formed in the composition at a sufficiently low temperature – dissolve.

20 The crystallization point of the peroxide in the composition can be determined by storing the cyclic ketone peroxide composition with and without the co-crystallizing compound at a pre-determined first temperature. If, after 1 hour (while stirring the composition), crystals have been formed, the composition is heated to a temperature that is 3°C higher. After about 6 hours it is checked 25 whether the crystals have dissolved. If they are not dissolved, the temperature of the composition is raised another 3°C and kept at that temperature about 6 hours. This process is repeated until a final temperature is reached at which all the crystals are dissolved. This final temperature is the crystallization point of the peroxide in the composition.

30 If in contrast no crystals have been formed after 1 hour at said first

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temperature, a very small amount of seeds of pure cyclic ketone peroxide is added to the composition (this is called seeding). After the addition of the pure peroxide, the composition is checked for crystals after 24 hours. In case no crystals were formed, the temperature of the composition is decreased 10°C. If 5 after the temperature decrease crystals are formed, the temperature is raised according the above procedure until all of the crystals are dissolved. However, if no crystals were formed, the temperature of the composition is decreased another 10°C, after which the composition is seeded again, and the procedure after seeding is repeated as described above until the crystallization point of the 10 peroxide is established.

For compositions comprising a co-crystallizing compound, the temperature is lowered to or below the temperature at which crystals are formed in the conventional composition (without the co-crystallizing compound). 15 Subsequently, the composition is seeded and stored for at least two days. Then, the temperature of the composition is raised until the solidified co-crystallizing compound has redissolved in such an extent that the composition is sufficiently clear to analyze any crystals present in the composition. This second temperature is preferably lower than the crystallization point of the 20 conventional cyclic ketone peroxide formulations (without the co-crystallizing compound).

If this second temperature is lower than the crystallization point of the composition, and crystals are observed, the composition is not considered to be safe and is not storage stable. If this second temperature is lower than the 25 crystallization point of the composition, and no crystals are observed, the composition is safe and storage stable and a composition according to the invention.

In case the crystallization point of the peroxide composition is below the second temperature, and no crystals are observed, the composition is considered safe 30 and storage stable and in accordance with the present invention. However, if

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the crystallization point of the peroxide composition is below the second temperature, and crystals are observed, the composition is considered not to be safe.

5 Next to the above described "crystallization point" test, the peroxide compositions according to the present invention should also pass the following conventional safety tests:

- the Deflagration test (deflagration),
- the Time Pressure test (deflagration),

10 - the Koenen test (heating under defined confinement),

- the Pressure Vessel test (heating under defined confinement), and
- the Thermal Explosion Vessel test (heating under defined confinement).

Passing these tests means a "medium" or "low" rating in the heating under defined confinement tests and a "no" or "yes, slowly" rating in the deflagration

15 tests. The final hazard rating, for which the most severe rating in any one of the tests is used, must be "medium" or "low."

The safety tests and corresponding criteria are documented in the "United Nations Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria".

20 The composition of the present invention can be prepared by producing the cyclic ketone peroxide in the phlegmatizer according to the invention or after its preparation the cyclic ketone peroxide can be dissolved in the phlegmatizer of choice. Preferably, the cyclic ketone peroxide is manufactured directly in the 25 phlegmatizer and/or co-crystallizing compound in accordance with the present invention. The manufacture of a composition having a high active oxygen content is advantageous with respect to an efficient use of the reactor and the reagents.

The invention composition may be further diluted with the phlegmatizer and/or

30 the co-crystallizing compound of choice in order to comply with regulations for

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storage and transport, in particular in the case of bulk quantities, which are stored and transported in intermediate bulk containers and tanks. The phlegmatizer and/or co-crystallizing compound may be added to the peroxide at any time, thus before, during or after the preparation of the peroxide as long as

5 it is added before storage.

When cyclic ketone peroxides are prepared starting from a suitable ketone, usually a mixture of peroxides is formed which predominantly consists of the trimeric and dimeric forms. However, the compositions may also comprise some linear as well as some tetrameric and higher oligomeric cyclic structures.

10 Preferably, the cyclic ketone peroxide consists essentially of trimer and dimer in amounts as defined above.

The ratio between the various forms, notably the trimer/dimer ratio, mainly depends on the reaction conditions during the preparation, and the person skilled in the art is referred to WO 96/03397 for obvious variations in the

15 reaction conditions that can be carried out in order to influence this ratio. If so desired, the reaction mixture may be separated into the individual cyclic ketone peroxide compounds. However, in order to avoid laborious purification procedures, the composition of the invention will typically contain some dimeric structures next to trimeric ones, as defined earlier.

20 Preference for certain compositions or individual compounds may depend on differences in physical properties or requirements in the application of the peroxides, e.g. storage stability, half-life time versus temperature, volatility, boiling point, solubility, etc.

25 The composition of the present invention may optionally contain certain additives as long as these additives do not have a significant negative effect on the safety, transportability and/or storage stability of the formulation. As examples of such additives may be mentioned: antiozonants, antioxidants, antidegradants, U.V. stabilizers, coagents, fungicides, antistatic agents,

30 pigments, dyes, coupling agents, dispersing aids, blowing agents, lubricants,

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process oils, and mould-release agents. These additives may be employed in their usual amounts.

Normally, such additives will be added to the phlegmatized cyclic ketone peroxide formulation in accordance with the present invention shortly before the 5 formulation is used in a polymerization or polymer modification process.

The present invention also relates to the use of these compositions in radical (co)polymerization processes, polymer modification processes such as controlled rheology polypropylene processing, and other reactions involving 10 peroxides, like the synthesis of certain chemicals. By applying the cyclic ketone peroxide formulations according to the present invention, less phlegmatizer is introduced into the various applications, either allowing higher peroxide loads in the process or generating polymeric products with improved properties and containing fewer impurities derived from the phlegmatizer used.

15

The present invention is illustrated by the following examples.

EXAMPLE

20 Over a period of 60 minutes 14.1g 70% aqueous hydrogen peroxide was added to a mixture of 20.8g methyl ethyl ketone, 22.5g Isopar® M, 0.75g Paraffin® and 19.2 g 50% aqueous sulfuric acid, while stirring the mixture. The obtained mixture was stirred for another 60 minutes, after which the temperature was increased to 35°C and kept at this temperature for 60 minutes. Subsequently, 25 the organic layer was separated and neutralized with 15.0g of an aqueous solution of 4N sodium hydroxide, after which the neutralized layer was stirred for 30 minutes. This neutralized organic layer was washed twice with water, dried with 1.0g of magnesium sulfate dihydrate and filtered. The thus obtained peroxide composition (i.e. dried organic layer) is diluted with Isopar M so as to 30 obtain a composition having a total active oxygen content of 7.5 wt%, based on

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the total weight of the peroxide composition. 97% of the total active oxygen content is attributable to cyclic methyl ethyl ketone peroxides.

This composition was observed to be storage stable and safe, whereas the  
5 same formulation without the paraffin was not.

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## CLAIMS

1. A cyclic ketone peroxide composition comprising one or more crystallizing peroxides, optionally one or more conventional phlegmatizers (diluents), and one or more co-crystallizing compounds which solidify in the final formulation at a solidification temperature above the crystallization temperature of the crystallizing peroxide.
- 5 2. A composition according to claim 1 wherein the cyclic ketone peroxide is methyl ethyl ketone peroxide.
- 10 3. A composition according to any one of claims 1 and 2 wherein a co-crystallizing compound is selected from a group consisting of a cyclic hydrocarbons, non-cyclic hydrocarbons, esters, cellulose esters, hydrogenated castor oil and fumed silica.
- 15 4. A composition according to any one of the preceding claims wherein the composition is a liquid at the recommended storage temperature or the handling temperature.
- 20 5. Use of compositions according to any of the preceding claims in radical (co)polymerization processes, and in polymer modification processes.

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## ABSTRACT

The present invention relates to a cyclic ketone peroxide composition comprising one or more crystallizing peroxides, optionally one or more conventional phlegmatizers (diluents), and one or more co-crystallizing compounds which solidify in the final formulation at a solidification temperature above the crystallizing temperature of the crystallizing peroxide. These peroxide compositions have an improved storage stability and safety. The invention also pertains to the use of compositions according to this invention in (co)polymerization and polymer modification processes.

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